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REPORT No. O - 5/53

Studies of Organic Phosphors for Use as Scintillation Counters

Part II.

Growth from Solution and Thermal Expansion
of Stilbene Crystals

S. K. Hutchinson

Ruth Lapage

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ATOMIC WEAPONS RESEARCH ESTABLISHMENT

WEAPONS AND ASSEMBLY DIVISION

REPORT NO. O-5/53

SCW BRANCH

Studies of Organic Phosphors for Use as Scintillation
Counters

Part 2. Growth from Solution and Thermal Expansion
of Stilbene Crystals
(Part 1. Report O-4/53)

S. K. Hutchinson
Ruth Lapage

September, 1953.

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1. Introduction

Studies of growth of large crystals of stilbene from the melt⁽¹⁾ indicated a high thermal expansion coefficient for stilbene. Moreover, from the ease with which the stilbene crystals cracked under slight thermal strain it seemed likely that they would possess a considerable degree of thermal anisotropy.

A study, therefore, has been made to determine the principal coefficients of thermal expansion of stilbene crystals. From a knowledge of these coefficients of expansion the orientation of growth of crystals grown from the melt can be readily determined by measuring linear thermal expansions of small cubes cut from large single crystals.

Stilbene crystals are monoclinic and thus one of the three principal coefficients of thermal expansion lies along the diad axis. To obtain the remaining two coefficients it is necessary to measure thermal expansions in three directions in a plane normal to the diad axis (i.e. the (010) plane).

The linear thermal expansions have been determined by the interferometric method originally devised by Pizeau. For this purpose stilbene crystals were grown from solution since, apart from the ease with which the crystallographic axes can be determined on such crystals in contrast to those grown from the melt, the crystals possess natural parallel faces which allow determination of thermal expansions in the (010) plane without further treatment of the crystal. For the expansion along the diad axis it is however necessary to prepare parallel faces by grinding.

From the values of the principal coefficients of thermal expansion the cubical expansion coefficient has been obtained and confirmed by measurements carried out using a dilatometer. In addition, cubical expansions have been determined with the dilatometer over a temperature range 0° - 120°C, and the temperature coefficient of the cubical coefficient of thermal expansion obtained.

2. Growth from Solution

The successful growth of crystals of reasonable size of many water-soluble compounds (2,3) from solution suggested that the method might be applied to stilbene from organic solvents. After some preliminary experiments acetone was chosen as the solvent. The solubility of stilbene in acetone was measured and found to be 86 gms./litre at 28°C. and 143 gms./litre at 43°C..

The crystals were grown by immersing the seeds in a saturated solution and allowing the solution to evaporate or cool slowly. The experiments were carried out in a 5-litre beaker which was placed in an unstirred water thermostat heated at the bottom. This method of heating prevented the growth of unwanted crystals on the bottom of the beaker since crystals, formed either on top of the solution during evaporation or within the solution, on sinking to the bottom of the beaker were redissolved in the warmer layer there. The top of the lipless beaker was ground flat and covered with a glass plate through which passed a stirrer. This plate reduced evaporation in the cooling experiments and, furthermore, the rate of evaporation could be controlled in the evaporation experiments by varying the diameter of the stirrer. The stirrer consisted of a brass rod (of about $\frac{1}{4}$ inch diameter) on to which were soldered six cross-pieces, about

$\frac{1}{4}$ inch apart, of 16 S.W.G. copper wire. Short lengths of Smtoflex tubing were forced over these copper wires and the seed crystals (twelve), of about 2 mm.² cross-section, forced well into the other end. This form of holder has the advantage that when the crystals have grown the tube is slipped off the wire and can then be pulled out of the crystal without danger of cracking it.

The solution was prepared by dissolving a weighed amount of purified stilbene⁽¹⁾ in the acetone at a higher temperature than required followed by reduction in temperature with stirring until saturation was reached. For the evaporation experiments the temperature used was about 28° - 30°C.; in the cooling experiments the solution was cooled from approximately 45° to 25°C..

When the solution was saturated the seed crystals were introduced and the temperature raised 0.5°C. so that any crystals, either formed due to slight supersaturation or adhering to the seeds or stirrer, were dissolved. The solution was stirred at about 20 revs./minute and the direction of rotation was reversed every minute. This was effected by a relay system reversing the current in a D.C. motor, the relays being operated by a thyatron valve-condenser circuit (See appendix for circuit diagram and operation).

The solution was allowed to evaporate by about 50 cc. a day or was cooled 4°C. a day. A Synclock motor (1 rev./hour) was used to rotate the magnet controlling the position of the contact wire in a contact thermometer to give the required rate of cooling. When the crystals had grown to the required size the stilbene remaining in solution was precipitated by the addition of water at the same temperature as the acetone. The solution was then cooled to room temperature and the crystals removed. The addition of water to the solution prevented the crystals cracking by local cooling due to acetone evaporation from their surfaces on removal of the crystals from the solution.

The size of the apparatus used in these experiments limited the size of the crystals grown to about 2 cm. x 2 cm. x 1 cm.. The quality of these crystals was generally good, except for an occasional tendency for veils to appear in the early growth immediately round the seeds. Such crystals were not used for the expansion measurements. Some typical crystals are shown in Figure 1. Tests have shown that these crystals behave identically with those grown from the melt in scintillation counters. Crystals twinned along the (001) plane occasionally during growth from solution and hence were rejected.

3. Interferometric Determination of the Principal Coefficients of Thermal Expansion

3.1 Experimental

The method employed was similar to that first devised by Fizeau⁽⁴⁾ and later extended and improved by Tutton⁽⁵⁾. A description of a less elaborate apparatus than Tutton's was given by Scott⁽⁶⁾ for determination of thermal expansions where the highest accuracy was not required. This same apparatus has been used for the work described here although some small modifications have been made to it.

The apparatus is shown diagrammatically in Figure 2. Basically, the method consists of following the movement of parallel equidistant interference fringes, formed between an optical flat attached to the specimen surface and a stationary optical flat, as the specimen expands. The distance between neighbouring fringes corresponds to $\frac{\lambda}{2}$ of the monochromatic light employed. Thus by counting the number of fringes passing a reference point, such as the cross-wires of a telescope, for a measured temperature difference, the coefficient of thermal expansion can be readily derived. Small corrections need to be made to allow for the expansions of the optical flats and the tripod legs.

To minimise the correction for expansion of the tripod legs an invar tripod was used. The crystal specimen (of thickness 0.5 cm. - 1.5 cm. approximately) was mounted as shown between two fused quartz optical flats. Rigidity of mounting was obtained by cementing the crystal to the two flats with very thin films of butyl methacrylate monomer (containing benzoyl peroxide as polymerisation accelerator) which were allowed to polymerise overnight in a warm oven (about 40°C.). With the stationary optical flat in position resting on the tips of the three legs of the tripod, the legs were adjusted to obtain the interference fringes using the procedure described in detail by Scott(6). This, briefly, is by obtaining an almost parallel film of air between the flats using a small piece of cover slip and then adjusting the tripod legs until parallel interference fringes observed by reflection of monochromatic sodium light appear between the two flats. When the fringes are obtained, the positions of the legs are firmly fixed by locking nuts.

The invar tripod was supported on a steel stand carrying two glass flats as shown in Figure 2. These flats served to reflect the incident and reflected light from the interferometer. The rest of the optical system is clearly shown in Figure 2. To avoid undue strain of the eyes of the operator, the telescope eyepiece was removed and the image of the fringes in the telescope objective observed through a low power microscope.

The stand carrying the invar tripod was placed in a well-lagged air oven. This oven was fitted with a stirrer and heated by mat heaters placed at the sides and back behind thin asbestos shields. The shields were raised about $\frac{1}{4}$ inch from the floor of the oven to allow circulation of the air. To eliminate direct radiation of heat on to the interferometer, this was placed in a small 3-sided asbestos box, the open side facing the glass window of the oven. The oven could be thermostatted by a contact thermometer in conjunction with a relay (Sunvic) operating the mat heaters. The front window of the oven was lagged with a square of $\frac{1}{4}$ inch thick asbestos sheet possessing an aperture in the centre for the incident and reflected light and for allowing thermometers in the oven to be read. Since the temperature of the crystal is required two calibrated thermometers were placed both sides of the tripod with their bulbs close to the crystal. The mean of the two temperatures measured was taken as the temperature of the crystal. Expansions were determined both by heating and cooling the crystal. Rates of heating and cooling were controlled by a Variac in series with the heaters.

The usual procedure for carrying out the measurements was to do a heating run during one day, to leave the oven thermostatted at about 45°C. overnight and then cool during the next day. Before the start of either a heating or cooling run temperatures were recorded over a period of half an hour and the mean temperature obtained. The variation in temperature near room temperature was negligible but at about 45°C. the variation was $\pm 0.2^\circ\text{C}.$ The rates of heating and cooling were of the order of 5°C./hour at the beginning, slowing down to about 2°C./hour for the final stages.

3.2 Results and Discussion

From the count of the number of fringes (N) passing the cross-wires in the telescope for a temperature change of ΔT , a value $N/\Delta T$ was calculated. This was found to increase steadily during a run, reaching a constant after about 15°C. change in temperature. Theoretically $N/\Delta T$ should be a constant from the beginning of the run but since the crystal temperature lagged behind the air temperature during the first stages of both the heating and cooling runs, the temperatures recorded were too great. With the oven used by Scott(6) $N/\Delta T$ fell to a constant due to the interferometer receiving heat by direct radiation from the heater (an unscreened Robertson lamp) and thus being initially hotter than the air.

The constant $N/\Delta T$ is a measure of the change in width of the air film per °C. Thus $N/\Delta T \times \frac{\lambda}{2}$ (where λ = wavelength of monochromatic Na light) is the

change in units of length of the air film due to the relative expansions of the crystal, the quartz flats and the invar legs of the tripod. If Q is the expansion of the flats per $^{\circ}\text{C}$. and I that of the invar legs, then the expansion due to the crystal is

$$\frac{N}{\Delta T} \times \frac{\lambda}{2} + I - Q.$$

Hence if H is the thickness of the crystal, the coefficient of linear expansion

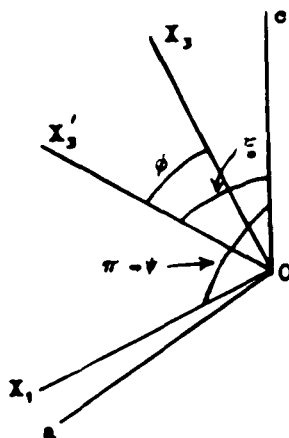
$$\alpha = \frac{\frac{N}{\Delta T} \times \frac{\lambda}{2} + I - Q}{H}.$$

Values of linear coefficients of thermal expansion of invar (0.09×10^{-5}) and fused quartz (0.042×10^{-5}) were taken from the literature(7). Since the corrections due to these expansions are small highly accurate values are not required. Variation of ± 10 per cent in the values leads to variation of α well within the limits of accuracy of measurement of $N/\Delta T$.

Stilbene forms monoclinic crystals (Figure 3). In the monoclinic crystal system, one principal coefficient of thermal expansion lies along the diad (or b) crystallographic axis and the remaining two lie in the (010) plane, normal to the b axis. Neither of these coefficients need be coincident with either the a or c axes. To determine the values of these two coefficients and their positions relative to the a or c axes, measurements of thermal expansion in three arbitrary directions in the (010) plane are necessary(8). The stilbene crystals possessed three well-developed forms (403), (001) and (201) parallel to the diad axis and thus could be used without further treatment for the expansion measurements in the three directions in the (010) plane. To determine the expansion coefficient along the b axis it was necessary to grind flat each end of the crystal on fine emery paper to prepare parallel faces.

From the three measurements in the (010) plane the two coefficients and their positions were determined in the following manner(8).

FIG. 4.



In Figure 4, OX_1 and OX_2 are the expansion axes, a and c the crystallographic axes, and OX_3 one of the arbitrarily measured directions. If α_{11} , α_{33} and α'_{33} are the expansion coefficients along OX_1 , OX_2 and OX_3 respectively then

$$\frac{(1 + \alpha'_{33})^2}{(1 + \alpha_{33})^2} \cos^2 X'_3 \delta X_3 + \frac{(1 + \alpha'_{33})^2}{(1 + \alpha_{11})^2} \cos^2 X_1 \delta X'_1 = 1$$

By neglecting second powers of α_{11} , α_{33} and α'_{33} it can be shown that

$$\alpha'_{33} = \alpha_{11} \sin^2 \phi + \alpha_{33} \cos^2 \phi$$

Therefore

$$\begin{aligned} \alpha'_{33} &= \frac{1}{2} (1 - \cos 2\phi) \alpha_{11} + \frac{1}{2} (1 + \cos 2\phi) \alpha_{33} \\ &= \frac{\alpha_{11} + \alpha_{33}}{2} - \frac{(\alpha_{11} - \alpha_{33})}{2} \cos 2\phi. \end{aligned}$$

Since

$$\pi - \psi - \frac{\pi}{2} = \xi - \phi,$$

then

$$\phi = -\frac{\pi}{2} + (\xi + \psi)$$

and

$$\alpha'_{33} = \frac{\alpha_{11} + \alpha_{33}}{2} + \frac{\alpha_{11} - \alpha_{33}}{2} (\cos 2\xi \cos 2\psi - \sin 2\xi \sin 2\psi).$$

Let

$$\begin{aligned} A &= \frac{(\alpha_{11} + \alpha_{33})}{2} \\ B &= \frac{(\alpha_{11} - \alpha_{33})}{2} \cos 2\psi \\ C &= -\frac{(\alpha_{11} - \alpha_{33})}{2} \sin 2\psi \end{aligned}$$

then

$$\alpha'_{33} = A + B \cos 2\xi + C \sin 2\xi.$$

From three values of α'_{33} and the corresponding angle ξ , A, B and C can be obtained.

Now $-\tan 2\psi = C/B$, $\alpha_{11} = A + B/\cos 2\psi$ and $\alpha_{33} = A - B/\cos 2\psi$

Hence ψ , α_{11} and α_{33} can be readily derived.

The angle ξ is obtained from the geometry of the crystal. Angles between the crystal faces for stilbene have been given by Groth(9) and have been confirmed by the authors using a Unicam optical goniometer.

Table I lists the measured expansions along the various directions for several crystals.

Table I

Expansions (cm. $\times 10^{-5}$ /cm./ $^{\circ}$ C.)

Crystal	Along b axis	Normal to 001	Normal to 201	Normal to 403
1		12.46 12.63 13.04		
2		12.87 13.09 13.21		
3		12.92 13.39		
4	7.47		6.27	
5	7.27 7.65 7.65 7.44 7.38 7.38			
6			6.53 6.47 6.60	
7				8.12 8.12 8.44 8.59 8.59
8				8.51 8.51
Mean Values	7.47	12.98	6.47	8.41
Standard Deviations	0.13	0.26	0.26	0.20

By substituting the three mean values of expansion in the (010) plane with their appropriate angles of inclination to the C axis in the equation

$$\alpha'_{j3} = A + B \cos 2\xi + C \sin 2\xi ,$$

values

$$A = 9.27$$

$$B = 2.61$$

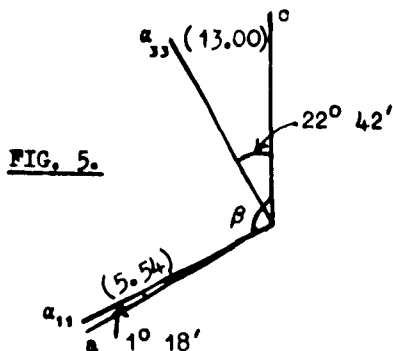
$$C = 2.66$$

are obtained.

Hence

$$\alpha_{11} = 5.54, \alpha_{33} = 13.00, \psi = 67^{\circ} 12'.$$

Figure 5 shows the positions of these two expansion axes relative to the crystallographic a and c axes. The angle β for stilbene is $114^{\circ} 6'$ (9).



In view of the limits of accuracy of the measurements the angle $1^{\circ} 18'$ can be neglected, the expansion axis a_{11} , thus being regarded as coincident with the a crystallographic axis. The major expansion axis for stilbene is therefore normal to the (001) plane.

Table II summarises the thermal expansion data for stilbene crystals.

Table II

Principal Expansion Axis	Linear Expansion $\times 10^{-5}/^{\circ}\text{C.}$	Standard Deviation	Cubical Expansion $\times 10^{-5}/^{\circ}\text{C.}$
a	5.54	0.26	26.0
b	7.47	0.13	
c' (normal to 001)	13.00	0.26	

Stilbene crystals cleave readily along the (001) plane indicating that the intermolecular forces normal to this plane are the weakest in the crystal. This view is borne out by the X-ray data(10). The unit cell, containing four molecules, is $a = 12.35$, $b = 5.70$, $c = 15.92$. The greatest separation between the molecules occurs between the layers parallel to the (001) plane. Hence it would be expected that the greatest expansion coefficient would lie along an axis normal to the (001) plane. It is of interest to note here that the principal expansion axes coincide with those of diamagnetic susceptibility(11), and furthermore the greatest anisotropy of susceptibility lies in a direction normal to the (001) plane.

4. Measurement of Cubical Expansion by Dilatometer

4.1 Experimental

The dilatometric method consists of totally immersing the solid in a liquid in a small bulb and measuring the change in position of the meniscus of the liquid along a capillary tube attached to the bulb for a particular temperature change. To calculate the cubical expansion of the solid the expansion of the liquid is required, and, furthermore, a small correction must be made to allow for the expansion of the apparatus.

It is convenient to use a liquid in which the solubility of the solid is negligible otherwise corrections must be made for the solubility coefficient of the solid. The liquid must completely wet the solid phase since air bubbles, trapped between solid particles, will give too high an expansion coefficient. The apparatus must therefore be evacuated before or during filling with the liquid. Up to temperatures of about 70°C. water was found to be a suitable liquid but above this temperature the formation of air bubbles during the experiment gave erratic results. Above 70°C. mercury was used satisfactorily.

The experimental details are as follows.- About 2 grams of stilbene were placed in a small cylindrical bulb (7 cm. long \times 0.6 cm. diameter)

connected by a B7 joint to a capillary tube of approximately 0.5 mm. bore, the two halves of the apparatus being held rigidly together by extension springs. The joint was sealed by the minimum amount of a silicone grease. The capillary tubes (Chance's 'Veridia' Precision Bore Tubing), calibrated with a small mercury bead, were selected for use where the cross-sectional area varied by only ± 0.5 per cent. The stilbene samples, either grown from the melt or from solution, were in the form of small lumps ($\frac{1}{8}$ inch to $\frac{1}{4}$ inch cubes approximately). These lumps were carefully selected to avoid cracks or internal flaws which might lead to entrapped air bubbles. The apparatus was evacuated and the liquid added. The vacuum (approximately 1 mm. pressure) was maintained until air bubbles no longer appeared in the column of liquid in the capillary tube, even on vigorous shaking of the apparatus.

For measurements up to 95°C., the dilatometer was mounted in a large glass water thermostat (about 40 litre capacity). The thermostat heaters were controlled by a contact thermometer and relay. By suitable adjustment of the heater currents and rates of stirring a constancy of temperature of $\pm 0.005^\circ\text{C}$. at 25°C. and $\pm 0.015^\circ\text{C}$. at 95°C. was obtained. At 120°C. a small metal bath containing liquid paraffin was used, the temperature control being approximately $\pm 0.1^\circ\text{C}$.

The change in position of the meniscus of the liquid in the capillary tube was measured with a cathetometer. About 15 minutes was allowed at each temperature for the dilatometer and contents to reach the bath temperature. Measurements of the changes in the position of the meniscus were reproducible to within ± 0.002 cms.. For the 120°C. bath, a stem correction had to be applied for the part of the capillary protruding above the liquid level. The temperature taken for this correction was the mean of temperature at the meniscus level and the paraffin surface.

4.2 Results

The results were calculated from the equation

$$\alpha' = \frac{[(l_1 - l_2) - wyAT]A\rho}{\Delta T.m}$$

where α' = apparent expansion coefficient (i.e. uncorrected for the expansion of the apparatus).

$(l_1 - l_2)$ = change in position of meniscus for a temperature change ΔT .

m = weight of stilbene.

w = weight of liquid.

ρ = density of stilbene at the minimum temperature for each temperature range.

A = cross-section of the capillary tube.

y = expansion in cms./gram /°C. of the liquid up the capillary tube.

y was determined for each apparatus by measuring the expansion of the liquid alone. The difference between the measured expansion of the liquid and that calculated from the literature data(7) gave a correction factor α'' for the expansion of the dilatometer. Several measurements were made to determine α'' and the mean value taken.

The true coefficient of cubical thermal expansion is thus $\alpha = \alpha' + \alpha''$.

Table III summarises the results obtained.

Table III

Temperature Range °C	Liquid	$\alpha' \times 10^{-5}$	No. of Experiments	$\alpha \times 10^{-5}$	Standard Deviation
0-35	Water	1.3	7	24.3	0.4
25-45	Water	1.3	5	25.9	0.4
	Mercury	1.2	3	25.9	-
35-70	Water	1.1	7	27.5	0.6
70-95	Mercury	1.3	3	28.7	-
95-120	Mercury	1.0	4	33.4	0.4

The value of the cubical expansion coefficient for the temperature range 25° - 45°C. obtained with the dilatometer is 25.9×10^{-5} and is therefore in good agreement with the value 26.0×10^{-5} derived from the interferometric measurements.

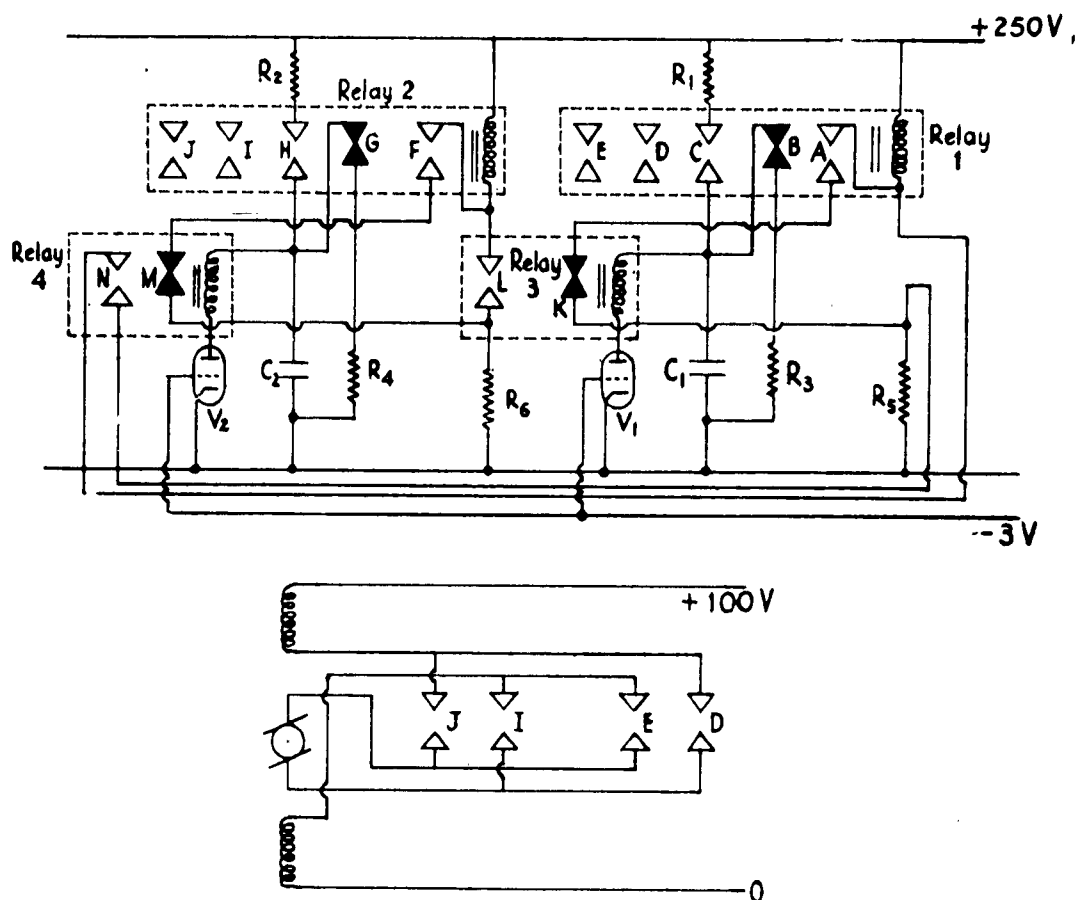
The results given in Table III are plotted in Figure 6 and from the linear graph the temperature coefficient of cubical expansion has been obtained. The cubical expansion of stilbene can be defined by the equation

$$v_t = v_0 (1 + \alpha t + \beta t^2)$$

where $\alpha = 2.43 \times 10^{-4}$ and $\beta = 8 \times 10^{-7}$.

APPENDIX

Circuit Diagram and Operation of Relay System



The cycle of operation is started by closing relay 4 manually for a short time (i.e. N closed, M opened). Current flows in relay 1 and contacts A, C, D and E close and B opens. The contacts E and D of relay 1 are connected, as shown in the lower diagram, to the field and armature of the D.C. motor. The condenser C_1 charges through R_1 and after 1 minute the Thyatron valve V_1 strikes and closes relay 3 for a short time. Thus relay 1 is opened by contacts K and relay 2 closed (i.e. F, H, I, J) by contacts L. The contacts I and K of relay 2 are connected to the D.C. motor in a reverse manner to contacts E and D. C_2 is charged through R_2 and after a minute the Thyatron valve V_2 strikes, opening F, H, I and J of relay 2 by means of contacts M and closing relay 1 by contacts N thereby initiating another cycle of operations.

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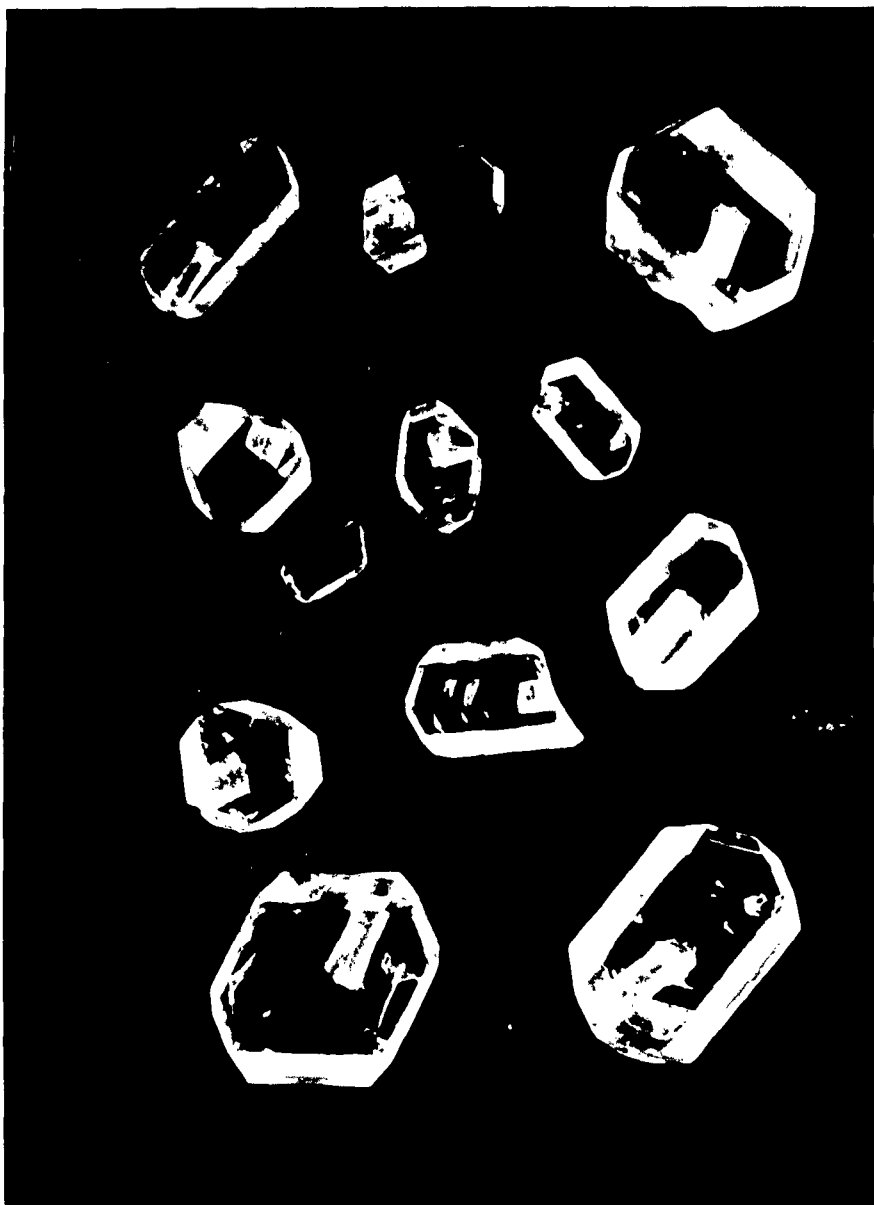
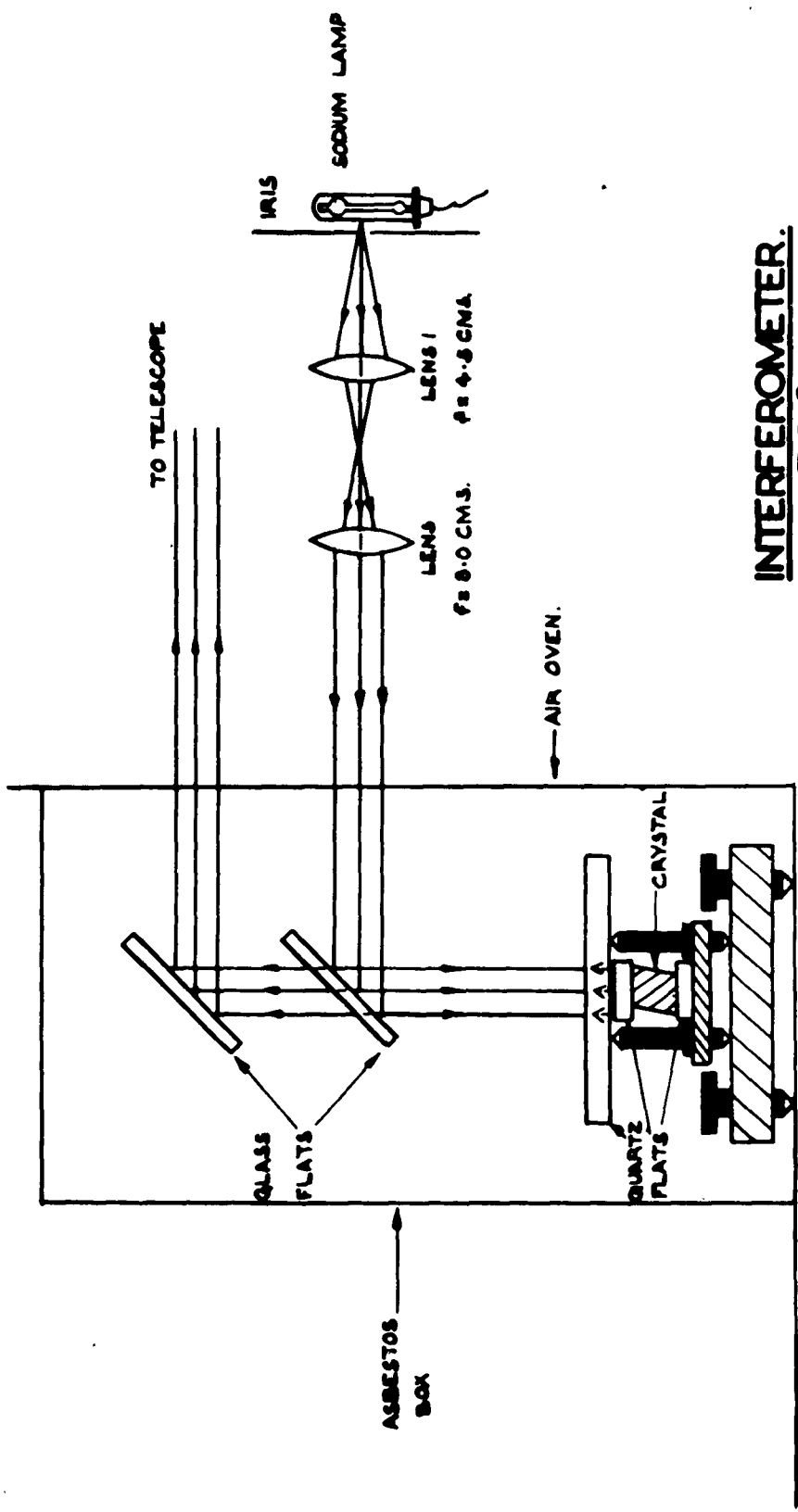
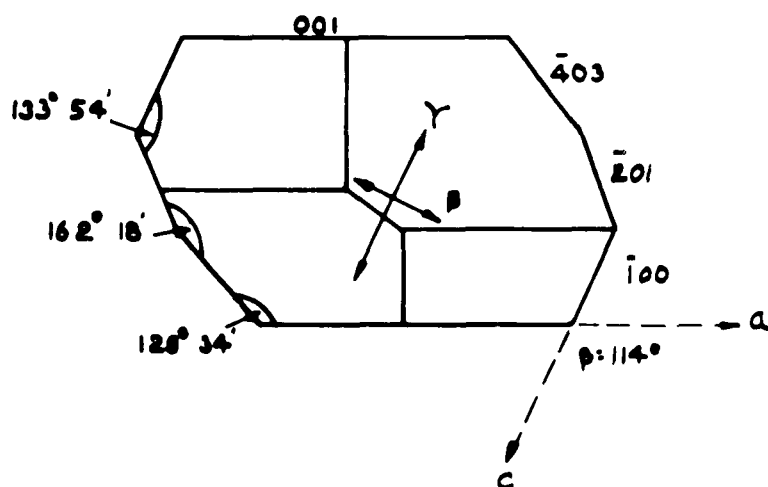
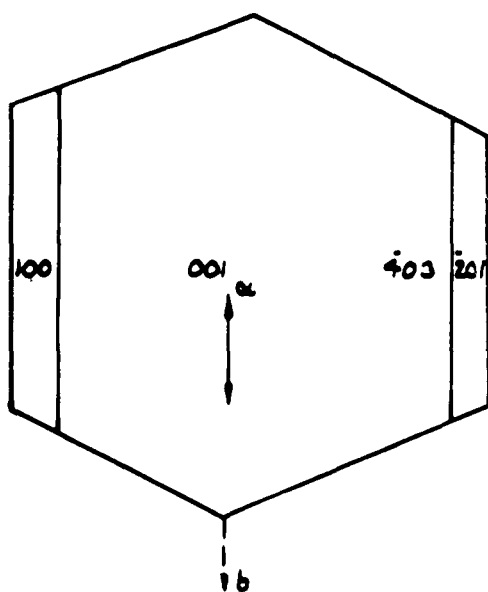
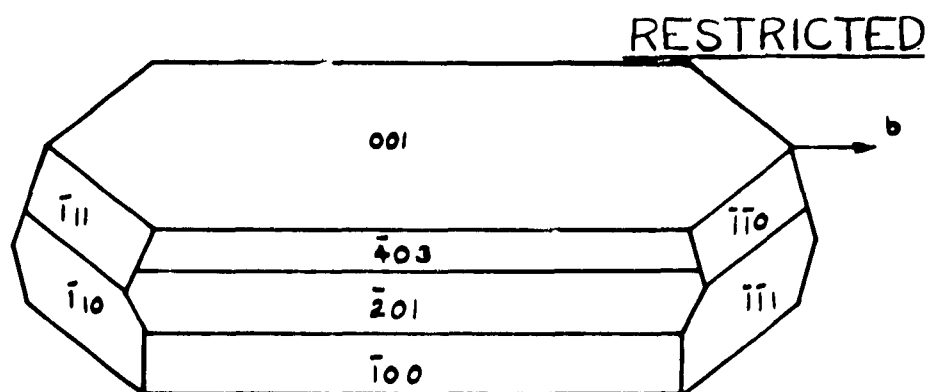


FIG. 1. STILBENE CRYSTALS ($\times 2$).



INTERFEROMETER.
FIG:2.



TYPICAL STILBENE CRYSTAL.

(FROM ACETONE.)

FIG. 3.

RESTRICTED

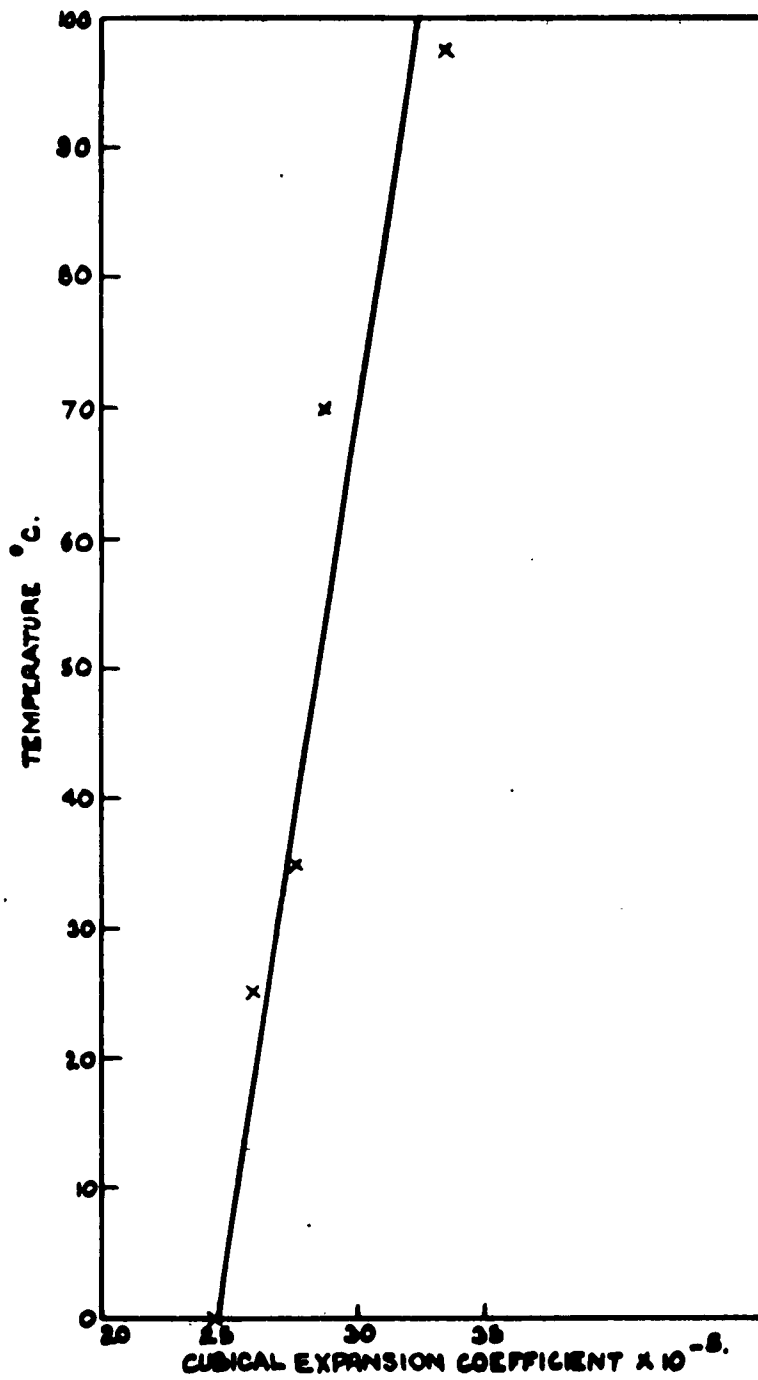


FIG:6.



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